

25 September 1963

PURCHASE EXHIBIT F-63-A
JET FUEL, LOW VOLATILITY

1. SCOPE

1.1 SCOPE - This exhibit covers one grade of aircraft turbine and jet engine fuel.

2. APPLICABLE DOCUMENTS

2.1 The following standards, of the issue in effect on date of invitation for bids, form a part of this exhibit:

STANDARDS

Federal

Federal Test Method Standard No. 791

Lubricants, Liquid Fuels, and
Related Products; Methods of
Testing

Military

MIL-STD-290

Packaging, Packing and Marking
of Petroleum and Related Pro-
ducts.

2.2 Other publications. The following documents form a part of this exhibit to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS PUBLICATIONS

ASTM Standards on Petroleum Products and Lubricants

D270 Standard Method of Sampling Petroleum and Petroleum Products

(Copies of ASTM publications may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pennsylvania)

3. REQUIREMENTS

3.1 Materials. The fuel shall consist completely of hydrocarbon compounds except as otherwise specified herein, or as duly approved by the procuring activity.

3.2 Chemical and physical requirements. The chemical and physical requirements of the finished fuel shall conform to those listed in section 3.

TABLE I - CHEMICAL AND PHYSICAL REQUIREMENTS AND TEST METHODS

Requirements	Value	Test Method	
		Fed Std 791	ASTM Standard
Distillation:	1)	1001	D86
Initial boiling point, °F.	375 min.		
Fuel evaporated, 10 per cent at °F.	400 min.		
Fuel evaporated, 20 per cent at °F.	To be reported		
Fuel evaporated, 50 per cent at °F.	420 min.		
Fuel evaporated, 90 per cent at °F.	500 max.		
End point at °F.	550 max.		
Residue, vol. per cent at °F.	1.5 max.		
Distillation loss, vol. per cent at °F.	1.5 max.		
Gravity, °API - min. (sp. gr. max.)	47 (0.793)	401	D287
Gravity, °API - max. (sp. gr. min.)	53 (0.767)	401	D287
Existent gum, mg/100 ml	7.0 max.	3302	D381
Sulfur, total, per cent by weight	0.1 max.	5201	D1266
Mercaptan sulfur, per cent by weight or doctor test	0.005 max.	5204	D1219 or D1323
	Sweet	5203	D484
Freezing point, °F.	-40 max.	1411	D1477
Net Heat of Combustion, BtU. per lb.	18,900 min. ²⁾		
Luminometer Number	100 min.		D1740
Viscosity, centistokes at -30°F.	15 max.	305	D145
Aromatics, vol. per cent	5 max.	3703	D1319
Copper strip corrosion, ASTM classification	No. 1 max. ³⁾	5325	D130
Flash point, °F.	150 min.	1102	D93
Water separator index	85 min.	3256	
Thermal Stability	4)		
Change in pressure drop in 5 hours in Hg	5 max.		
Preheater deposit code	2 max.		
Vapor Pressure	5)		
psia at 300°F.	2.7 max.		
psia at 500°F.	45 max.		

1) A condenser temperature of 32° to 40° shall be used for the distillation.

2) This test is not included in Federal Test Method Standard No. 791 or ASTM standards. See paragraph 4.6.1.

3) To be performed in accordance with paragraph titled "Tests at 212°F. (100°C.) for Volatile Materials" of ASTM D130-56.

4) This test is not included in Federal Test Method Standard No. 791 or ASTM standards. See paragraph 4.6.2.

5) Value shall be calculated in accordance with para 4.6.3 of this specification.

3.3 Additives. The additives listed herein may be used singly or in combination in amounts not to exceed those specified. The type and amount of each additive used shall be reported.

3.3.1 Antioxidants. The following active inhibitors may be blended separately or in combination into the fuel in total concentration not in excess of 8.4 pounds of inhibitor (not including weight of solvent) per 1,000 barrels of fuel (9.1 gm/100 U.S. gallons, 2.4 mg/liter or 109 mg./imp. gal.) in order to prevent the formation of gum:

- a. 2,6 - ditertiary butyl - 4 methylphenol
- b. N, N' - Dissecondary butyl paraphenylene diamine
- c. 2,4 - dimethyl - 6 tertiary - butylphenol
- d. 2,6 - ditertiary - butyl phenol
- e. Mixed tertiary butyl phenols composition 75 percent 2,6 - ditertiary butyl-phenol 10 to 15 percent 2,4,6 - tritertiary butyl phenol.
- f. 10 to 15 percent ortho-tertiary butyl phenol

3.3.2 Permitted Additives: Additives other than those listed in paragraph 3.3.1 will be permitted only with the authorization of the procuring activity.

3.4 Workmanship. The finished fuel shall be visually free from undissolved water, sediment or suspended matter and shall be clean and bright at the ambient temperature or at 70°F.

3.4.1 Odor. The odor of the fuel shall not be nauseating or irritating. No substances of known dangerous toxicity under usual conditions of handling and use shall be present.

1. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Classification of tests. For acceptance purposes, individual lots shall be subjected to all tests and other requirements cited in section 3.

4.3 Inspection lot.

4.3.1 Bulk lot. An indefinite quantity of a homogeneous mixture of material offered for acceptance in a single isolated container.

4.3.2 Packaged lot. An indefinite number of 55-gallon drums or smaller unit packages of identical size and type offered for acceptance and filled with a homogeneous mixture of material.

4.4 Sampling. Each bulk or packaged lot of material shall be sampled for verification of product quality and compliance with Standard MIL-STD-290 as applicable, in accordance with Method 8001 of Federal Test Method Standard No. 791 (ASTM D270).

4.5 Inspection. Inspection shall be in accordance with Method 9601 of Federal Test Method Standard No. 791 (ASTM D270).

4.6 Test Methods. Tests, to determine conformance to chemical and physical requirements, shall be conducted in accordance with Federal Test Method Standard No. 791 or ASTM standards, using the applicable methods as listed in Table I, except for the following.

4.6.1 Net Heat of Combustion. The net heat of combustion shall be determined by use of the bomb calorimeter test described in appendix I of this exhibit.

4.6.1.1 Reported Data. The following data shall be reported: Net heat of comb., in BTU/lb.

4.6.2 Thermal stability. The thermal stability test shall be conducted using a CRC High Temperature Research Fuel Coker with reservoir fuel temperature maintained at 300°F during 5 hour operation at conditions of 500°F preheater temperature, 600°F filter temperature, and 6 lb per hour fuel flow rate.

4.6.2.1 Reported Data. The following data shall be reported.

a. Differential pressure in inches of mercury at 300 minutes, or time to a differential pressure of 5 inches of mercury whichever comes first.

b. Preheater deposit code rating at the end of the test.

Note: See Test Nr. 3464 of Federal Std No. 791 for information on Pressure Drop and Preheater Ratings using thermal stability test apparatus.

4.6.3 Vapor Pressure - Values shall be calculated using Figure 1. The 20% evaporated point reported using ASTM test D86 (Reference Table I) shall be used as a basis for this calculation.

4.6.3.1 Reported Data. The following data shall be reported:

a. The vapor pressure in pounds per square inch absolute at 300°F.

b. The vapor pressure in pounds per square inch absolute at 500°F.

Note: the procuring activity reserves the right to determine conformance to the 300°F vapor pressure requirement by using a reflux method outlined in appendix II of this specification.

5. PREPARATION FOR DELIVERY

5.1 Packaging, packing, and marking. Packaging, packing, and marking shall be in accordance with Standard MIL-STD-290.

6. NOTES

6.1 Intended use. The fuel covered by this exhibit is intended for use in engines other than reciprocating types.

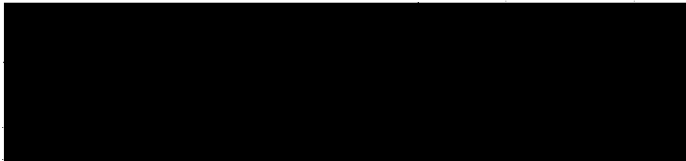
6.2 Ordering data. Procurement documents should specify the following:

a. Title and number of this exhibit.

b. Method of Delivery.

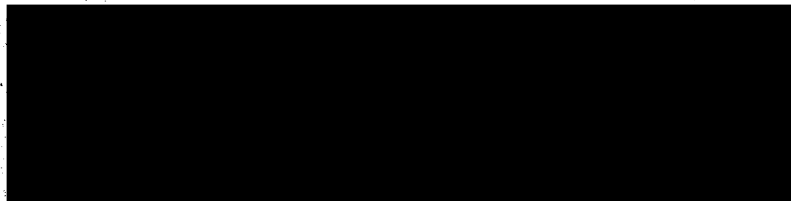
6.2.1 The material will be purchased by volume, the unit being a U.S. gallon at 15.5°F (15.5°C).

6.3 FORWARDING CERTIFICATE OF ANALYSIS - One copy of the certificate of analysis listing those items in Table I will be forwarded to the following organization for all fuel procured under this specification:



STATINTL

6.4 NOTICE TO CONTRACTING OFFICERS - The fuel covered by this exhibit is not intended for general procurement. It is a limited production item to be consumed only by systems using engines that require this product. The buyer should contact the following organization if clarification of requirement is required:



STATINTL

Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter

1. Introduction:

1.1 This method for heat of combustion is basically similar to ASTM Method D240 modified only to provide greater precision. With the development of supersonic jet fuels and their narrow specification ranges, the limits given in ASTM D240 of repeatability of 55 BTU per pound and reproducibility of 175 BTU per pound were not suitable.

2. Scope:

2.1 This method was primarily developed for supersonic jet fuels; however, it may also be used for other fuels, and volatile or non-volatile solids and liquids.

Summary:

3.1 Heat of combustion is determined in this method with an adiabatic calorimeter and by burning an accurately weighed sample in an oxygen bomb. The heat rise is measured by a resistance thermometer under closely controlled conditions. The heat of combustion is computed from temperature observations before, during, and after combustion with allowance for thermo-chemical and heat transfer corrections.

4. Definitions and Units:

4.1 The energy units in this method are defined as follows:

4.1.1 One calorie (International Steam Table Calorie) equals 4.1868 absolute joules.

4.1.2 One BTU (British Thermal Unit) equals 251.996 calories or 1055.07 absolute joules.

4.2 Heat of combustion, computed in calories per gram and converted to British Thermal Units per pound (one calorie per gram equals 1.8 BTU per pound).

4.3 Energy Equivalent (effective heat capacity or water equivalent) of the calorimeter is the energy required to raise the temperature one degree, expressed as calories per degree Centigrade.

4.4 Temperatures shall be recorded in ohms or other units; however, the same units must be used in all calculations, including standardization.

5. Apparatus:

5.1 Adiabatic Calorimeter - Series 1200 adiabatic oxygen bomb calorimeter, The Parr Instrument Company.

5.2 Automatic Temperature Controller - Model No. 2601, Parr Instrument Company.

5.3 Oxygen Bomb - Double valve oxygen bomb No. 1101, Parr Instrument Company. The internal volume is 360 mls.

- 5.4 Sample Cup - Stainless steel, E.H. Sargent and Company - weight of cup - 8 ± 1.0 grams.
- 5.5 Firing Wire - No. 45C10, Parr Instrument Company.
- 5.6 Platinum Resistance Thermometer - Leeds and Northrup, 25 ohm calorimetric type.
- 5.7 Mueller Resistance Bridge - Minneapolis Honeywell, Model 1551.
- 5.8 Galvanometer - Leeds and Northrup, Model 2430.
- 5.9 Thermometer - Parr Calorimetric, 0.05F subdivision.
- 5.10 Thermometer Reading Lens - Parr Instrument Company.
- 5.11 Analytical Balance - Mettler Instrument Corporation, sensitivity: 1 microgram.
- 5.12 Pellet Press - Parr Instrument Company, used to prepare Benzoic Acid in pellets weighing 0.9 to 1.1 grams.
- 5.13 Firing Circuit - A 12 volt alternating or direct current is required for ignition purposes with a pilot light in the circuit to indicate when current is flowing.

6. Reagents:

- 6.1 Oxygen - Commercial oxygen produced from liquid air.
- 6.2 Sodium Hydroxide - 0.0725N, 2.9 grams NaOH in 1 liter water standardized against Potassium acid phthalate using phenolphthalein as indicator.
- 6.3 Methyl Orange - pH range 3.1 to 4.4
- 6.4 Benzoic Acid standard - National Bureau Standards sample No. 39L.

7. Preparation of Apparatus:

- 7.1 Clean and polish cups with #600 grit paper, rinse in trichloroethylene, place in covered beaker and allow to dry. Always use forceps when handling cups.
- 7.2 Open water valve and turn on water heater.
- 7.3 Clean bomb, bucket and flask thoroughly.
- 7.4 Connect platinum resistance thermometer to Mueller bridge.
- 7.5 Plug in galvanometer.
- 7.6 Chill cold-water.

8. Standardization:

8.1 Weigh 0.9 to 1.1 grams of Benzoic Acid and transfer to pellet press and compress. Prepare at least 6 pellets each time and store in covered dish.

8.2 Determine energy equivalent of calorimeter using benzoic pellets by steps described in Procedure.

8.3 Determine and calculate the corrections for nitric acid and fused wire.

8.4 Calculate the energy equivalent by means of the following equation:
$$W = \frac{Hg + e_1 + e_2}{t}$$

Where:

W - energy equivalent of calorimeter in calories per ohm.

H - heat of combustion of standard benzoic acid, calories/gram.

g - weight of standard, in grams.

t - corrected temperature rise - ohms resistance.

e₁ - correction for heat of formation of nitric acid, in calories.

e₂ - correction for heat of combustion of firing wire, in calories.

8.5 Standardization tests should be repeated after changing any part of the calorimeter and occasionally as a check on both calorimeter and operating technique.

8.6 A minimum of 3 runs should be made to determine energy equivalent.

9. Procedure:

9.1 Preparation of Bomb -

9.1.1 Dry bomb and place 1 ml of water in bomb using syringe.

9.1.2 Dry bomb head and electrodes and place in stand.

9.1.3 Securely fasten 10 cm of firing wire to bomb head electrodes and fashion into "U" shape. Remove from stand and place bomb head into bomb.

9.1.4 Using a clean sample cup, weigh a sample of sufficient size which will produce a temperature rise equal to that produced by the combustion of 1.0 ± 0.1 grams of benzoic acid standard, (Approximately 0.56 grams). The volatility rate of each type of fuel must be pre-determined by measuring weight loss and time required to load the bomb. A correction for this loss is made on the final sample weight.

9.1.5 Place sample cup in holder and position firing wire just above the surface of sample. Sample cup should be tilted slightly to one side so that emerging flames do not impinge on electrodes.

9.1.6 Fit bomb head and turn screw cap down firmly by hand. Attach filling connection to bomb inlet valve and slowly admit oxygen to 30 atmospheres gage pressure at room temperature without purging air. During assembly, filling and handlings prior to filling, the bomb must be handled carefully in order not to disturb sample.

9.2 Calorimeter Water -

9.2.1 A 2000 ml volumetric flask is filled with distilled water and the temperature carefully adjusted to 76.2°F. At this temperature, the volume is accurately adjusted to the 2000 ml graduation. Extreme care must be exercised to insure precise temperature and volume adjustment.

9.3 Assembly of Calorimeter -

9.3.1 Thoroughly clean and dry calorimeter bucket and place in water jacket. Insert bomb in bucket and connect firing wire to Terminal.

9.3.2 Add calorimeter water to bucket and drain for at least 30 seconds. Close jacket cover, insert thermometers, thermistors and platinum resistance thermometer.

9.4 Automatic Temperature Controller -

9.4.1 Turn on the switch of the automatic temperature controller. The stirrer motor on the calorimeter is controlled by the same switch.

9.4.2 Use the manual control switch of the temperature controller to quickly bring the jacket temperature into close agreement with the bucket temperature.

9.4.3 Release manual switch and let the controller make any further fine adjustments. If necessary, make adjustments with the "balance" knob of the temperature controller to bring the jacket temperature in equilibrium with the bucket temperature. Check temperatures by use of glass thermometers and adjust jacket temperature.

9.5 Bridge Adjustment -

9.5.1 Check all connections and tighten. Connect the 4 leads of the resistance thermometer to the 4 binding posts marked C, c, t, T.

9.5.2 Connect a 2.5 volt battery to BA binding post and the galvanometer to the GA positions.

9.5.3 Insert plug firmly in position marked Ratio. Place commutator in neutral position (midway between N and R). Set plug switch X1 and X.1 switches on zero. Balance by adjusting the lower three dials until galvanometer exhibits a minimum deflection as the 0 key is held down - (approximate setting 395) now set dials X1 and X.1 on R and depress 0 key. If the galvanometer now exhibits the same deflection as before, the ratio is in proper adjustment.

9.5.4 Place all switches on zero. Turn commutator to N position, press 0 key, and balance the bridge using the "Adjust Zero" dial. The setting of the adjust zero dial must not be disturbed during measurements.

9.5.5 Transfer the three position plug to position marked "Measure". Set commutator on N position and place plug switches and dials to desired positions for measuring resistance.

9.6 Combustion of Sample -

9.6.1 Determine initial resistance reading by adjusting switches of Mueller Bridge until galvanometer is balanced. This reading is taken approximately 5 minutes after calorimeter has reached equilibrium. Record resistance reading.

9.6.2 Depress firing button for 6 to 8 seconds. Check temperature rise on glass thermometers. Allow approximately 7 minutes from the time of ignition to final resistance reading. These time intervals for reaching equilibrium and taking final resistance reading were accepted after many months of trial and error. Record final resistance reading.

9.7 Analysis of Bomb Contents -

9.7.1 Remove bomb from calorimeter - release pressure in such a manner that elapsed time shall not be less than one minute. Wash interior of bomb, electrodes, and sample cup with a minimum of distilled water, preferably less than 350 mls. Titrate washings with standard sodium hydroxide using methyl orange as indicator. Remove and measure unburned wire. Subtract from 10 cm. and record difference as wire consumed.

10. Calculations:

10.1 Temperature Rise - From resistance readings, using appropriate calibration chart, calculate temperatures. Using these values, calculate temperature rise in calorimeter as follows: $t = t_f - t_a$

Where:

t = corrected temperature rise

t_a = temperature when charge was fired

t_f = final equilibrium temperature

10.2 Gross Heat of Combustion - Compute by substituting in the following equation:

$$Hg = \frac{tW - e_1 - e_2 - e_3}{g}$$

Where:

Hg = gross heat of combustion, in calories per gram

t = corrected temperature rise

W = energy equivalent of calorimeter, in calories per ohm.

e_1 = thermochemical correction, correction for heat of formation of nitric acid in calories = milliliters standard alkali solution used in titration.

e_2 = thermochemical correction, correction for heat of formation of sulfuric acid in calories = $14 \times$ percent of sulfur in sample \times weight of sample in grams.

e_3 = thermochemical correction, correction for heat of combustion of firing wire = wire factor \times centimeters of wire consumed.

g = weight of sample, in grams.

10.3 Net Heat of Combustion - If the percentage of hydrogen is known, substitute in the following equation: $H_n = 1.8 Hg - 91.23 \times H$. If the percentage of hydrogen is not known, substitute in the following equation: $H_n = 4310 + (1.2951) (Hg)$

Where:

H_n = net heat of combustion, in BTU/lb.

H = hydrogen content, in percent.

PURCHASE EXHIBIT F-634A

APPENDIX II

Reflux Method
For Determination of Vapor Pressure of Liquids
Below Atmospheric Pressure

Introduction:

Vapor pressure measurement by the boiling point method or boiling point as a function of pressure is not a recent idea. The literature is prolific with various ramifications of this idea for use in determining the vapor pressure of pure compounds but provide cautious remarks when referring to mixtures of compounds. This report provides a description of the equipment and detailed procedure used for the determination of vapor pressure by the reflux method.

Description of Method:

An equilibrium condition of vapor evolution and refluxing of the evolved vapors is made under conditions of fixed vacuum (pressures below atmosphere) by applying heat to the test fluid. Equilibrium conditions exist when the temperature of the fluid and the temperature of the refluxing vapors approach each other and maintain a constant minimum temperature differential. The system pressure at which equilibrium refluxing occurs is regarded as the vapor pressure of the test fluid for the particular liquid temperature observed. Similar "boiling points" are obtained by gradually increasing the system pressure step-wise up to atmospheric pressure. By plotting liquid temperature against system pressure, a smooth curve is obtained from which vapor pressure below atmospheric pressure may be obtained at any desired temperature.

Description of Equipment:

Referring to the attached photograph, number XP 3314 and Schematic Figure 1, the following apparatus is required:

Apparatus:

1. Vacuum Pump
2. Powerstat
3. Magnetic stirrer, Teflon-coated stirring bar magnet.
4. Heating mantle, Glas-col 500ml
5. Flask, pyrex round bottom, 500 ml, short neck, Grinding No. 24/40 with thermometer well.
6. Glass grinding, Pyrex $\text{\textcircled{S}}$ No. 24/40.
7. Glass grinding, Pyrex, $\text{\textcircled{S}}$ No. 10/30.
8. Condenser, reflux, Friedrichs, $\text{\textcircled{S}}$ grinding No. 24/30 Pyrex.
9. Thermometer, mercury, Centigrade, $\text{\textcircled{S}}$ grinding No. 10/30, 75 mm. immersion - 10 to 250C.
10. Thermometer, mercury, Centigrade, 76 mm. immersion -10 to 260C.
11. Manometer, U tube, mercury.
12. Rubber tubing, vacuum.
13. Rubber tubing, medium wall.
14. Stopcocks, needle valve, Brass $\frac{1}{4}$ ".
15. Condenser, cold finger, size 10, 300 mm. Corning No. 91300.
16. Flash, Vacuum, Cylindrical.
17. Gauge, Vacuum, Manometer.

Procedure:

1. Sample preparation and analyses prior to vapor pressure determination.

Approximately 600 ml. of sample are introduced into a large separatory funnel, chilled to + 32F for 6 hours or over night. The lower 50 to 100 ml are drawn off, discarded and an ASTM distillation range made of a portion of the remainder according to ASTM D 86 method.

2. Vapor Pressure Determination.

The apparatus is assembled in accordance with Fig. 1 and checked for leaks. 300 to 350 ml. of the fuel prepared as stated in Item 1 above are introduced into the 500 ml. flask. Ice, dry ice, or other cold environments are used in the cold trap to protect the vacuum gauges and the pump from contamination as well as serving as an indication of proper reflux action in the system. Proper refluxing is indicated, among other things, when no fractions are trapped in the cold trap. Experience with this rig has shown that a few drops of liquid accumulate in the trap with each run. However, a run which shows more than 1.5 ml. should be invalidated.

Begin the stirring action in the flask and carefully evacuate to degas the fluid. This should be done at ambient temperature but no longer than 10 minutes. Slowly increase the temperature of the contents in the flask by varying the powerstat. Note that a bleed valve (Schematic, Fig. 1 as No. 14A) is provided so that a continuous stable vacuum is maintained, i.e., evacuation with air by-pass is maintained at all times but control of system pressure is made by adjusting the valve in the pressure hose line leading to the cold trap (Item No. 14B in Fig. 1). With proper adjustment of temperature and pressure, a reflux action is noted within the condenser. For reproducibility, it is desirable to obtain refluxing to the same height for each run, arbitrarily selecting the first to second internal spiral of the Friedrichs condenser. When refluxing reaches an equilibrium at this level, the temperature of the liquid as measured in the well and the temperature of the reflux vapor should closely agree and for practical operating purposes a limit of 2 degrees Centigrade differential should be considered maximum. Change system pressure, increase temperature, and repeat the temperature-pressure measurements over the entire pressure range up to atmospheric pressure.

Record temperature of the flask contents, vapor temperature and pressure at stable reflux conditions. Stable reflux conditions are those where the temperature as shown by the 2 thermometers, the system pressure, and the height of the reflux in the condenser do not change on 3 successive readings at 2 to 3 minute intervals. Correction of the pressure as shown by the manometer for variations in barometric pressure should be made.

3. Analysis following the vapor pressure determination.

Allow the fuel to cool and return to ambient temperature and pressure conditions. Remove or decant sufficient fuel to determine the distillation range according to ASTM D-86 method.

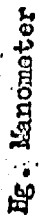


FIG. 1. SCHEMATIC-REFLUX VAPOR PRESSURE APPARATUS

TABLE I

